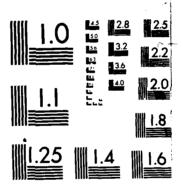
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REAL-TIME QUALITY CONTROL OF ANTIMISTING KEROSENE (AMK)

INTERIM REPORT BFLRF No. 187

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R.J. Mannheimer
Belvoir Fuels and Lubricants Research Facility (SwRI)
Southwest Research Institute
San Antonio, Texas

Prepared For

U.S. Department of Transportation Federal Aviation Administration Technical Center Atlantic City Airport, NJ 08405

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U.S. Army Belvoir Research and Development Center Materials, Fuels and Lubricants Laboratory Fort Belvoir, Virginia

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viscoelastic end corrections than AMK made with the original FM-9 polymer. Based on						
die-swell experiments, the outstanding rheological property of both antimisting fuels						
at high shear rates is the development of normal stresses that are 20 to 40 times						
larger than shear stresses. In phase II, experiments were conducted to determine the						
effect of polymer concentration and methods of blending on the rheological properties						
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measure the jet diameter and to eliminate the necessity of making end corrections. Simultaneous degradation/filtration experiments were also conducted in Phase II in which the critical filtration velocities with metal screens and paper filters were measured while AMK was being degraded. This approach has shown that the critical velocity for in-line blended AMK (age--30 minutes) is only slightly lower than for batch-blended AMK at the same specific degrader power. The high filtration ratio for in-line blended AMK (age--30 minutes) is caused by a decrease in the critical velocity and an increase in the rate of plugging that occurs after AMK is degraded. For square-mesh metal screens, a deformation rate was defined so that the onset of filter plugging by AMK is dependent of screen size. A similar parameter was defined for paper filters.

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LIST OF SYMBOLS

A	-	Area normal to flow, cm^2
A _o	-	Fraction of open area (metal screen), dimensionless
D	-	Diameter (inside) of tube, mm
$D_{\mathbf{N}}$	-	Nominal pore size (paper filter), μm
$D_{\mathbf{p}}$	-	Absolute pore size (metal screen), μm
ďj	-	Jet diameter (equilibrium), mm
F	-	Load, dynes
Н	-	Plate separation or gap, µm
L	-	Tube length, cm
n	-	Power law parameter, dimensionless
n '	-	Tube flow index $(n' = \frac{d \ln T}{d \ln 4V})$, dimensionless
$n_{\mathbf{F}}$	-	Tube flow index $(n^* = \frac{d \ln T}{d \ln 4V})$, dimensionless Torsional balance load index $(n_F = \frac{d \ln F}{d \ln \mathring{\gamma}_R})$, dimensionless
n _T	-	Torsional balance torque index $(n_T = \frac{d \ln T}{d \ln \hat{\gamma}_R})$, dimensionless
$\Delta \mathbf{P_T}$	-	Magnitude of total pressure drop, psi
$\Delta P_{\mathbf{v}}$	-	Magnitude of viscous pressure drop, psi
$\Delta P_{\mathbf{e}}$	-	End correction (Bagley), psi

LIST OF SYMBOLS (CONT'D)

R	-	Radius of tube or plate (torsional balance), mm
τ	-	Magnitude of shear stress at R, Pa
T	-	Torque (torsional balance), dyne cm
v	-	Average fluid velocity (tube or filter flow) or
		instantaneous plate velocity (torsional balance), cm/s
v_c	-	Critical velocity for filter, cm/s
ρ	-	Density, gm/mL
μ	-	Viscosity (Newtonian), dyn·s/cm ² or Poise
η _a	-	Apparent viscosity, dyn·s/cm ² or Poise
η _e	-	Effective viscosity, dyn·s/cm ² or Poise
Ω	-	Rotational speed, rad/s
ε	-	Porosity or void fraction, dimensionless
ν ₁ -ν ₂	-	Primary normal stress difference at R, Pa
N_{Re}	-	Reynolds number, dimensionless
[*] R	-	Shear rate at R, sec ⁻¹
n	_	Kinematic viscosity, centistokes

EXECUTIVE SUMMARY

This report discusses the results of a program to develop real-time quality-control tests for antimisting fuels. In Phase I of this program, a die-swell experiment is described in which the diameter of a jet is measured with a specially designed video-micrometer. A second rheological technique that utilizes the principle of torsional balance is also examined so that shear and normal stresses of antimisting fuels can be compared by two independent These experiments have established that antimisting kerosene (AMK) made with an improved version of the FM-9 polymer behaves similarly to AMK made with the original FM-9 polymer (i.e., the shear viscosity suddenly increases at a critical shear rate, reaches a maximum value, and then decreases at higher shear rates). However, the newer FM-9 polymer results in a lower critical shear rate, a higher maximum viscosity, and larger viscoelastic end effects. on die-swell, the most outstanding rheological properties of antimisting fuels at high shear rates (both AMK and 0.1 percent ARCO in Jet A) are large normal stresses that are 20 to 40 times larger than shear stresses. torsional-balance method was able to measure shear and normal stresses for 0.1 percent ARCO in Jet A at high shear rates, difficulties were encountered with In particular, quasi-static torsional-balance measurements indicated that the instantaneous viscosity of AMK at a shear rate of 2000 sec-1 is approximately the same as the maximum apparent viscosity measured with relatively short (200 < L/R < 400) tubes. However, the equilibrium viscosity, at this same shear rate, was less than one-third of this value. Since only equilibrium measurements can be used to calculate normal stresses, the torsional-balance method is unable to measure the instantaneous normal stresses developed by AMK before thixotropic breakdown has occurred. Furthermore, equilibrium torsional balance measurements for AMK exhibited a dependency on rotational speed that was not observed for the ARCO polymer. These difficulties prevented a comparison of normal stresses measured by die-swell and torsional-balance for AMK; however, in the case of the ARCO polymer, the normal stresses calculated from torsional-balance measurements were three to four times higher than the die-swell method.

In Phase II, die-swell experiments were continued, and two major improvements were incorporated into the prototype die-swell rheometer that was built for the Federal Aviation Administration: 1) an optical die-swell tester that measures the jet diameter (d_i) automatically, and 2) differential pressure taps that eliminate the need for making end-corrections (ΔP_{e}). Capillary tube experiments, conducted with the original die-swell rheometer at low shear rates, showed that $\Delta {f P_e}$ becomes important at the critical shear rate and that $\Delta {f P_e}$ is generally larger for smaller diameter tubes. Before correcting the total pressure drop (ΔP_T) for ΔP_e , the effective viscosity showed a strong dependence on both L/R and R; however, after correcting the data for ΔP_e , the viscosity was independent of tube size. Experiments that utilized differential pressure taps showed that a residence time of 3 seconds at a shear rate near 2000 \sec^{-1} resulted in a significant reduction in the viscosity of AMK and that thixotropic effects can be avoided by using relatively short (200 \leq L/R \leq 500) tubes. The maximum apparent viscosity, which occurred close to 2000 sec⁻¹, exhibited a strong linear dependence on polymer content and should be an important criterion for quality control.

Die-swell ratios for AMK increased with polymer concentrations, but the effect was not as large as expected. Furthermore, the normal stress function for AMK that was calculated from these measurements showed no measurable change with concentration. Since 0.35 percent FM-9 is a much more effective antimisting fuel than 0.25 percent, the invariance of the normal stress function over this same range of concentrations makes it of little use for quality control of AMK. This failure can be partly explained by inadequacies in the theory that relates die-swell to normal stresses. However, these experiments were limited to Reynolds numbers $(N_{\mbox{\scriptsize Re}}) > 100$ and much larger die-swell ratios are predicted for AMK if measurements are made at lower $N_{\mbox{\scriptsize Re}}$. This could be accomplished by using smaller diameter tubes and by working at shear rates near the maximum apparent viscosity (2000 to 3000 sec-1).

A simultaneous degradation/filtration test that measures the critical velocity of AMK (i.e., highest velocity that AMK can flow through a filter without plugging) while it is being degraded was also developed in Phase II. experiments were conducted by forcing AMK through a needle valve to produce the desired level of degradation and then through a small filter where ΔP is Results for square-mesh screens showed that the critical velocity measured. for in-line blended AMK, degraded 30 minutes after blending, was only slightly lower than the critical velocity for batch-blended AMK that was degraded at the same specific power. Furthermore, in both cases, the rate of pressure rise at velocities well above the critical was so low (e.g., at V = 2Vc it was only 0.05 psi/min) that no difficulties would be expected for short-term operations such as take-off, even if the critical velocity were exceeded. While the critical velocity increases with screen size, if measurements for different size screens are expressed in terms of V_c/D_pA_o , where D_p and A_o are the pore size and fraction of open area, respectively, the onset of filter plugging by AMK at fixed specific power is a constant. This is an extremely important result in that measurements for one screen size should be able to predict the critical velocity for any filter component provided it is a square-mesh screen. The effect of increasing specific degrader power is to increase the critical velocity (or equivalently V_c/A_0D_p). While there appears to be a relatively high threshold of 1000 psi or 6.7 kWs/L below which the critical velocity is only slightly higher than for undegraded AMK, it appears possible to increase the critical velocity to a value that will provide satisfactory filter performance at a specific power of less than 27.6 kWs/L with AMK at ambient temperatures.

Simultaneous degradation/filtration experiments have shown also that paper filters are much more sensitive to plugging by AMK than square-mesh metal screens. In particular, at the same specific power, the critical velocity for batch-blended AMK with a nominal 80-µm paper filter is approximately 1/20 of the critical velocity with a 74-µm square-mesh screen. While the complex geometry of paper filters is more difficult to characterize than square-mesh metal screens, if the critical velocities for different paper filters are expressed in terms of $V_{\rm c}/D_{\rm N}\varepsilon$, where $D_{\rm N}$ is the nominal pore size and ε is the porosity, the onset of filter plugging by AMK at a fixed specific power is a constant.

INTRODUCTION

BACKGROUND.

Rheological Mechanisms of Antimisting Kerosene. Kerosene-type fuels that contain only small amounts (0.1 to 0.3 wt percent) of a high molecular weight polymer can greatly reduce the potential hazard of a catastrophic fire in an impact-survivable crash (reference 1). This remarkable behavior is attributed to the resistance that these modified fuels exhibit to the formation of small drops when they contact a high velocity air stream. Thus, the descriptive name of antimisting kerosene or AMK has been adopted. High molecular weight polymers that are used for turbulent drag reduction are also effective antimisting agents. However, antimisting fuels made with these polymers generally have poor fuel delivery characteristics with centrifugal jet-transfer pumps. This problem is due to elasticity rather than viscosity and it becomes progressively worse as the fuel temperature decreases. current AMK formulation is made with the Avgard (ICI Americas Inc. trademark) slurry which contains an improved version of the FM-9 polymer and a glycol/amine This additive was developed to provide satisfactory mist-fire carrier fluid. protection and to minimize problems associated with the rate of polymer dissolution in Jet A and poor fuel delivery at low temperatures.

Although a high extensional viscosity (100 to 1000 times higher than the shear viscosity) is the most likely mechanism by which dilute polymer solutions are able to resist atomization, attempts to measure the extensional viscosity of AMK have not been very successful (references 2 and 3). These negative results suggested that AMK was not highly elastic and emphasis was shifted to the fact that the shear viscosity of AMK suddenly increases when the shear rate exceeds a critical value (references 2 and 4). Unfortunately, rheological measurements of AMK with rotational viscometers also have not been very successful. particular, complex flow instabilities often terminate cone and plate experiments by the sample being expelled from the gap when the critical shear rate is exceeded (reference 2). Thus, other than the value of the critical shear rate, very little rheological information has been learned about AMK with rotational viscometers. On the other hand, experiments with capillary tubes have shown that the shear viscosity of AMK reaches a maximum value and then decreases at higher shear rates (reference 5). However, the maximum shear viscosity is not large enough to explain why AMK is able to resist atomization so effectively.

Despite the previously mentioned difficulties of measuring the extensional viscosity of AMK, it is evident that AMK develops large normal stresses when the critical shear rate is exceeded. This conclusion is based on the swelling of jets of AMK as they emerged from the tip of a capillary tube (reference 5). Although the phenomenon of die-swell is attributed to the relaxation of normal stresses, the quantitative relation between normal stresses and die-swell is quite complex. Nevertheless, the die-swell that has been observed for AMK in capillary tube experiments suggests that the extensional viscosity of AMK becomes large at shear rates above the critical and is probably the primary rheological mechanism for antimisting.

Quality Control of AMK. Quality control tests that relate to the mechanism of antimisting are needed to assure that AMK will provide a predictable degree of mist-fire protection. Furthermore, in order for AMK to burn like Jet A in turbine aircraft engines and to flow through conventional aircraft fuel filters, the molecular weight of the FM-9 polymer must be reduced by a degrader. The current quality control tests that deal with flammability protection and intentional degradation are the "orifice cup" and "filtration ratio."

The orifice cup test has been developed by ICI and is highly specific to the characteristic shear thickening of AMK at the critical shear rate. The volume of AMK that flows through a small orifice in 30 seconds is reported as the orifice cup number (OC - mL/30 seconds). Although the length of the orifice is too short to be an accurate viscometer, typical OC values provide an estimate of the viscosity of AMK at a shear rate above the critical (reference 5). This high shear rate viscosity is a better quality control measurement for AMK than the low shear rate viscosity; however, the extensional viscosity would provide the best measure of antimisting quality. Unfortunately, the requirement of high strain rates makes the measurement of extensional viscosity for AMK impractical with existing technology. Since the extensional viscosity should be related to shear viscosity and normal stress functions, measurement of shear and normal stresses at high shear rates has been chosen for real-time quality control until a direct method of measuring the extensional viscosity of AMK can be devised.

The filtration ratio test developed by the Joint US-UK Technical Committee for AMK is currently the primary quality control test to measure intentional Basically, this test measures the time for a specific volume degradation. (96 mL) of fuel to flow between two timing marks on a vertically mounted glass tube (2.5 cm ID). A 16 to 18 μm Dutch weave screen is attached to the bottom of the tube, and the flow time for AMK relative to Jet A is reported as the filtration ratio (FR). For undegraded AMK, the FR is large (typically near 50); however, degradation can reduce it to close to 1.0. One problem with the filtration ratio test is that it may not discriminate between different levels of degradation for FR near 1.1 to 1.2. This problem has been shown to be due to a combination of relatively low rates of plugging with highly degraded AMK and the short duration (approximately 5 seconds) of the test (references 5 and 6). While the sensitivity of the filtration ratio test could be improved by either decreasing the flow area or by using paper filters instead of metal screens, the filtration properties of AMK have been shown to change with the time elaspsed after degradation. Furthermore, this time-dependent behavior is even more pronounced when AMK is degraded before the polymer has been fully solvated by Jet A. For example, when AMK is blended from Avgard, a satisfactory degree of mist-fire protection can be developed in less than However, this freshly in-line blended AMK appears to be more difficult to degrade than batch-blended AMK or in-line blended AMK that has had sufficient time (possibly several days) to fully equilibrate. Batch blending of AMK involves dispersing the FM-9 polymer in Jet A, slowly stirring in the glycol until the polymer is dissolved, and then adding the amine. blending of AMK is a more recent development in which the Avgard slurry is mixed directly into Jet A with a special blender developed by Jet Propulsion Laboratory. This conclusion regarding the apparent resistance of freshly in-line

blended AMK to degradation is based on the higher filtration ratio (FR > 10) than batch-blended AMK (FR = 1.2) degraded at the same specific power. Since the filtration properties of degraded AMK can change with time, there is some question as to the significance of the high FR. In effect, does it guarantee that freshly in-line blended AMK cannot be degraded enough to flow through typical aircraft filters? In order to answer this question, it is necessary to devise an experiment in which the filtration characteristics can be observed while AMK is being degraded.

PHASE I--SHEAR AND NORMAL STRESS MEASUREMENTS OF AMK AT HIGH SHEAR RATES

DIE-SWELL RHEOMETER.

Description of Experiments. The apparatus shown schematically in figure 1 was used to measure shear viscosity and die-swell at high shear rates. A pressure transducer was located in a 0.25-inch stainless steel tee immediately ahead of the capillary tube with the output of the transducer transmitted to a strip-chart recorder. A TV camera with a close-up lens was positioned near the outlet of the capillary tube to measure the diameter of the jet. This measurement was accomplished by aligning two cursor lines with the left and right edges of the jet displayed on the TV monitor (see figure 1). The resulting measurement was provided as a digital display on the video-micrometer. In general, both pressures and jet diameters were measured for several flow rates with different size tubes.

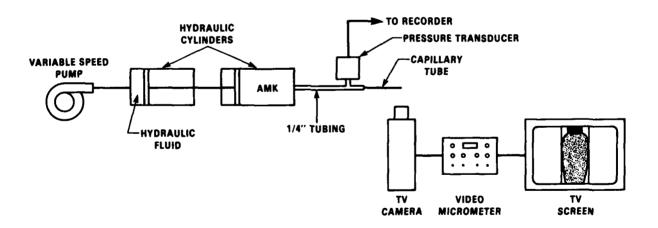


FIGURE 1. SCHEMATIC OF DIE-SWELL APPARATUS

While the majority of these measurements were made with AMK (i.e., 0.30 percent of the improved FM-9 polymer in Jet A that contains a glycol-amine carrier fluid), some experiments were conducted with a polymer manufactured by the ARCO Chemical Company that has been tested by the Federal Aviation Administration (FAA) and found to be an effective antimisting additive for Jet A. The ARCO additive was supplied to Southwest Research Institute (SwRI) by the FAA at a concentration of 0.1 percent polymer in Jet A. Only batch-blended AMK was used in Phase I; however, in Phase II, experiments were also conducted with "in-line" blended AMK using Avgard.

DISCUSSION OF RESULTS.

Shear Viscosity. The steady laminar flow in a tube of a material whose viscosity depends only on the shear rate (i.e., thixotropic time effects are negligible) must be a unique Function of the consistency parameters $\tau = R\Delta P/2L$ and 4V/R, where R is the inside radius of the tube, L is the length of the tube, ΔP is magnitude of the pressure drops and V = Q/A is the average velocity (reference 8). The quantity $\tau = R\Delta P/2L$ is the magnitude of the shear stress at the wall of the tube for any material. However, 4V/R or equivalently 8V/D (where D = 2R) is the shear rate at the wall $(\mathring{\gamma}_R)$ only for a special class of fluids (i.e., Newtonian). In all other instances, the shear rate is given by:

$$\mathring{\gamma}_{R} = \frac{3 + 1/n!}{4} \frac{8V}{D} \tag{1}$$

where

$$n' = \frac{d \log R\Delta P/2L}{d \log 8V/D}$$
 (2)

is the flow index. It is important to note that n' describes the shear dependency of a material. For example, $0 \le n' \le 1$ indicates that the viscosity is a decreasing function of the shear rate (i.e., shear thinning), while n' > 1 indicates that the viscosity is an increasing function of the shear rate (i.e., shear thickening) and n' = 1 indicates that the viscosity does not change with shear rate (i.e., Newtonian).

The apparent viscosity of a material is defined by the ratio of the wall shear stress to the wall shear rate:

$$\eta_{a} = \frac{\tau}{\dot{\gamma}_{R}} \tag{3}$$

Similarly, an effective viscosity can be defined simply by:

$$\eta_{e} = \frac{\tau}{4V/R} \tag{4}$$

Although the effective viscosity is not equal to the apparent viscosity, except for n'=1, it must not depend on R or L. In other words, if experimental measurements of pressure drop and flow rate are expressed in terms of consistency parameters, they should not show any dependence on R or L. However, data for AMK show a definite dependence on L in which the shear stress, at constant 4V/R, decreases with increasing L (figure 2).

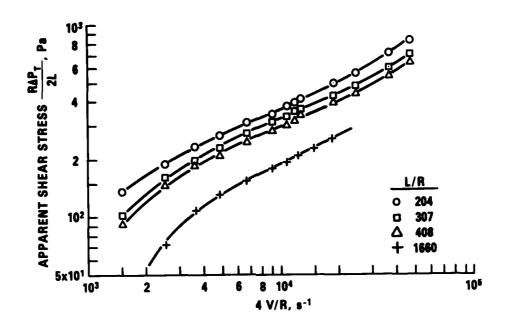


FIGURE 2. CONSISTENCY PARAMETERS FOR AMK BASED ON TOTAL PRESSURE DROP (R = 0.51 mm)

The anomalous dependency of the shear stress on L in figure 2 could be caused by the thixotropic changes in the flow-induced structure of AMK and the increase in residence time with tube length. In addition, ΔP_T , used in calculating the wall shear stress, has been assumed to be equal to the pressure measured in the 0.25-inch tee, i.e., $\Delta P_T = P - 0 = P$ (figure 1) less a correction for kinetic energy ($\Delta P_{Ke} = 1.1_{\rho} V^2$) that was usually negligible. More importantly, this procedure of estimating ΔP_T neglects the high resistance of viscoelastic materials to the accelerative flows at the ends of the tube (reference 9). It is important to point out that earlier experimental results for AMK made with the FM-9 polymer indicated that viscoelastic end effects were too small to detect for $4V/R < 7000~{\rm sec}^{-1}$ (reference 5). However, the newer FM-9 polymer results in a lower critical shear rate (1000 sec $^{-1}$ compared to 3000 sec $^{-1}$ for FM-9) and also appears to be more viscoelastic; consequently, the dependency on L shown in figure 2 could also be caused by viscoelastic end effects. The

Bagley method for determining the magnitude of these end effects utilizes a plot of ΔP_T versus L. The resulting relationship between ΔP_T and L should be linear with a positive intercept at L = 0 that is the pressure correction ΔP_e . The pressure drop associated with viscous dissipation in the tube is then calculated by ΔP_V = ΔP_T - ΔP_e .

While the Bagley method (reference 9) should not apply to thixotropic materials such as AMK, P_T is a linear function of L for a limited range of tube lengths (figure 3). More importantly, when these values of ΔP_e in figure 3 are subtracted from ΔP_T , the shear stress no longer shows a dependency on L for 200 < L/R < 400 (figure 4). On the other hand, the lower values of the shear stress for the longest tube (L/R = 1660) in figure 4 indicate that the effective viscosity of AMK was decreased by the longer residence time in this tube. This conclusion is supported by rheological measurements that will be discussed in the next section (Torsional-Balance) and in Phase II, in which the shear viscosity of AMK at 2000 sec^-1 is shown to decrease significantly in a time frame of 3 seconds. Furthermore, this is the approximate residence time for AMK in the longest tube at this shear rate (t = L/V = 4(L/R) \div 4V/R = 4(1660)/2000 sec^-1 = 3.3 sec).

The complex shear dependency of AMK is illustrated by the slope of the tangent line to the flow curve (i.e., the flow index n') for 200 < L/R < 400 in figure 4. In particular, in the range of 1500 sec^{-1} < 4V/R < 2500 sec^{-1} , n' > 1 indicates that the apparent viscosity is increasing. Actually, this shear thickening extends to values of 4V/R very close to $1000~{\rm sec}^{-1}$, but these measurements were not made for the data in figure 4. At higher shear rates, n' < 1 indicates that the viscosity is decreasing with increasing shear rate. Consequently, the shear viscosity of AMK reaches its maximum value of 36 mPa·s at $4V/R = 2500 \text{ sec}^{-1}$ (i.e., in figure 4 at $4V/R = 2500 \text{ sec}^{-1}$, n' = 1 and n_a = 90 Pa \div 2500 sec⁻¹ = 36 mPa·s). It is interesting to note that this maximum apparent viscosity is very close to the viscosity (30 to 33 mPa·s) calculated for OC values of 1.8 to 2.0 mL/30 seconds that are typical for AMK made with the improved FM-9 polymer (see reference 5 for details of this calculation). Furthermore, 4V/R associated with OC values of 1.8 to 2.0 mL/30 seconds are in the same range (2000 to 2500 sec⁻¹) as the shear rate associated with n' = 1 in figure 4. However, the very short orifice that is used in this test (L/R = 5) and the fact that no viscoelastic end corrections were applied to the pressure head make the reason for this close agreement difficult to explain.

The data in figure 5 summarize the results of experiments with different tubes in which both L and R were varied with L/R kept in the range of 200 to 400. While these results show some scatter (this is most likely due to the difficulty of estimating ΔP_e), there appears to be no systematic dependency on either R or L for flow data that have been corrected for ΔP_e . However, the uncorrected data showed a strong dependency on both L and R. The dependency on R resulted in higher viscosities for smaller tubes. This result can be explained by the fact that at the same value of 4V/R, ΔP_e increases with decreasing R. This larger ΔP_e can be explained in terms of the larger contraction ratios associated with the smaller values of R.

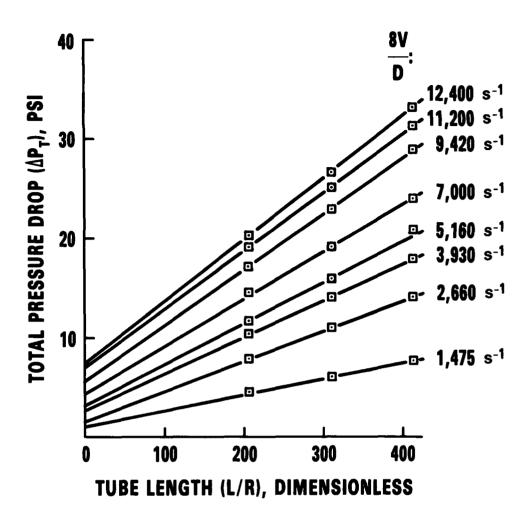


FIGURE 3. EFFECT OF TUBE LENGTH ON TOTAL PRESSURE DROP FOR AMK (R = 0.51 mm)

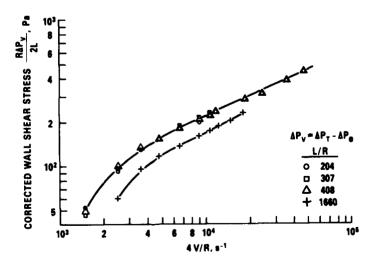


FIGURE 4. CONSISTENCY PARAMETERS FOR AMK CORRECTED FOR END EFFECTS (R = 0.51 mm)

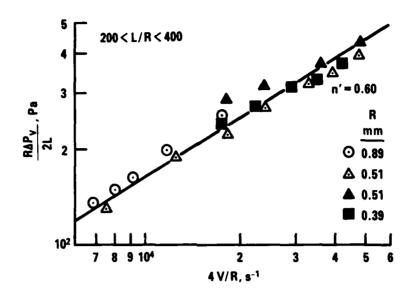


FIGURE 5. SHEAR DEPENDENCY OF AMK IN DIFFERENT TUBES

In order to calculate normal stresses from die-swell measurements, a reasonably accurate estimate of the shear dependency as a function of 4V/R is needed. Over the range of 4V/R for which die-swell measurements were made $(7000~{\rm sec}^{-1}<4V/R<50,000~{\rm sec}^{-1})$, the shear dependency of AMK is represented by a constant value of n' = 0.6. Unlike AMK, the drag-reducing polymer exhibited very little shear dependency over the range of conditions that corresponded to the die-swell experiments (i.e., for R = 0.39 mm, and 3000 ${\rm sec}^{-1}<4V/R<9000~{\rm sec}^{-1}$, and n' = 0.95). Because of this small degree of shear dependency, these results were not verified for different values of R. In fact, except for the large end corrections, even with very long tubes (figure 6), determining the shear dependency of the drag-reducing polymer was a relatively simple task.

The die-swell ratio (d₁/D) of Newtonian liquids was measured as Die-Swell. a function of $N_{Re} = DV\rho/\mu$ for different size tubes (figure 7). below 100, a strong dependence of d_{i}/D on D was observed in which d_{i}/D increased with decreasing D. For Newtonian liquids, di/D should, at most, depend on NRe (reference 10); consequently, some extraneous factors must have influenced Since all the tubes used in these experiments had the these measurements. same outside diameter, the wall thickness necessarily increased as D (inside Thus, wetting of the tip of the capillary tubes could diameter) decreased. enlarge the diameter of the jet. Furthermore, this wetting would have a proportionately larger effect with the smaller diameter tubes. This explanation is supported by the fact that the jet appeared to emerge from the outside edges of the tube at N_{Re} < 100. At N_{Re} > 100, this anomalous behavior was no longer evident. However, the limiting value of d_i/D (0.93 \pm 0.02) was higher than the theoretical value of 0.866 provided by Metzner and co-workers (reference Similar results have been reported for both die-swell (reference 10)

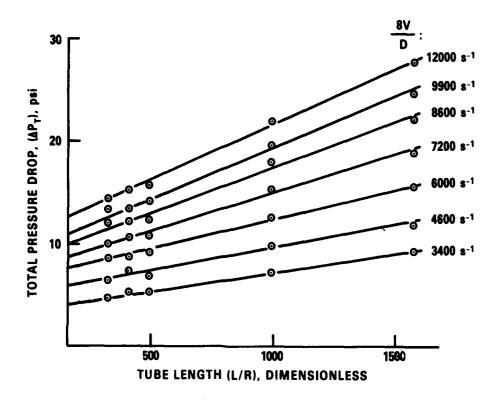


FIGURE 6. EFFECT OF TUBE LENGTH ON TOTAL PRESSURE DROP FOR 0.1 PERCENT ARCO IN JET A (R = 0.39 mm)

and the equivalent jet-thrust experiment (reference 11) in which d_j/D remained higher than the theoretical value. Metzner and co-workers (reference 10) have discussed some possible explanations and implications of these results.

Die-swell data for 0.1 percent ARCO and AMK are summarized in table 1. It should be mentioned that the Reynolds number, based on the apparent viscosity, was greater than 100 for all the data in table 1. These data, together with the measured shear dependencies, were used to calculate the normal stresses (figure 8) from Equation 5 developed by Metzner and coworkers (reference 12):

$$v_1 - v_2 \Big|_{R} = \frac{\rho D^2 (8V/D)^2}{64n!} \left\{ (n!+1) \left(\frac{3n+1}{2n+1} \right) - \left(\frac{D}{d_j} \right)^2 \left[n!+1 + \frac{d \log D/d_j}{d \log 8V/D} \right] \right\}$$
 (5)

where n is the power-law index and n' is the flow index. The term (3n+1)/(2n+1) in Equation 5 assumes that the velocity distribution can be approximated by a power-law fluid which was the case for the range of 4V/R in figure 8. The largest single difference between AMK and 0.1 percent ARCO in Jet A is the much higher shear viscosity of AMK at the same shear rate. While it is evident that the low shear viscosity of 0.1 percent ARCO in Jet A (3 mPa·s) can not be the reason for its excellent mist-fire protection, the higher viscosity of AMK (17 mPa·s) at $(17 \text{ mPa$

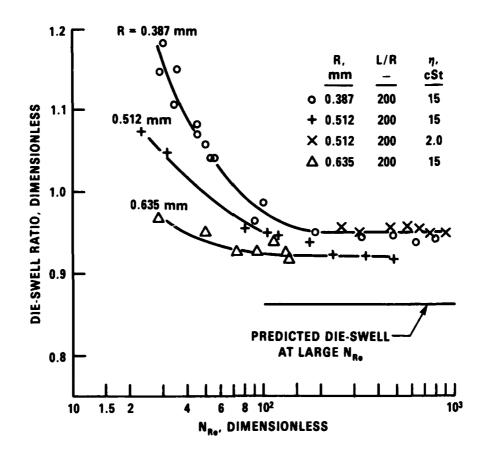


FIGURE 7. EFFECT OF TUBE SIZE AND REYNOLDS NUMBER ON THE DIE-SWELL OF NEWTONIAN LIQUIDS

The shear and normal stress behavior for the 0.1 percent ARCO in Jet A showed very little scatter and no effect on L/R. However, the normal stresses were significantly higher for the larger diameter tube in figure 8. While the shear behavior of AMK showed no significant dependence on R or L/R, the normal stresses exhibited large variations (this is indicated by the thickness of the error bands); consequently, no effect of R or L/R is apparent for the two smaller values of R. However, for R = 0.89 mm, the calculated normal stresses of AMK were larger, and the experimental scatter was reduced. This last statement can be partly explained by an improvement in the experimental technique. In particular, alignment of the tube with the optical axis of the TV camera was found to be important. Therefore, an alignment jig was used for R = 0.89 mm, 200 < L/R < 400. It is expected that less scatter would have been observed if this procedure had been used for the other tubes.

TORSIONAL-BALANCE RHEOMETER.

Description of Experiments. The operating principles of a torsional-balance rheometer have been previously discussed by Binding and Walters (reference 13) and are illustrated in figure 9. Two parallel plates of radius (R) are

TABLE 1. DIE-SWELL MEASUREMENTS OF ANTIMISTING FUELS AT $\rm N_{\mbox{\scriptsize Re}} > 100$

$\left(\frac{4V}{R}\right)^{10^{-3}}$		d _j /D	
sec ⁻¹	L/R = 200	300	400
	0.1 Percent ARCO	(D = 0.78 mm)	
3.3	1.48	1.50	1.48
4.5	1.47	1.48	1.47
5.7	1.46	1.46	1.46
7.0	1.45	1.45	1.45
8.3	1.43	1.43	1.43
9.6	1.42	1.42	1,41
	0.1 Percent ARCO	(D = 1.02 mm)	
4.2			1.28
5.4			1,26
6.0			1.25
7.3			1.24
8.5			1.24
	AMK (D =	0.78 mm)	
22.2	1.64	1.48	1.41
28.8	1.36	1.29	1.24
35.4	1.25	1.19	1.18
42.4	1.18	1.12	1.12
49.0	1.12		1.10
	AMK (D =	1.02 mm)	
18.2	1.17	1.20	1.15
23.9	1.05	1.08	
			1.05
35.9	1.04	1.02	1.09
47.6	1.01	1.11	1.08
	AMK (D =	1.78 mm)	
6.8	1.20	1.18	1.13
8.0	1.16	1.15	1.12
9.1	1.14	1.14	1.08
11.7	1.10	1.10	1.06
17.4	1.06	1.05	1.04

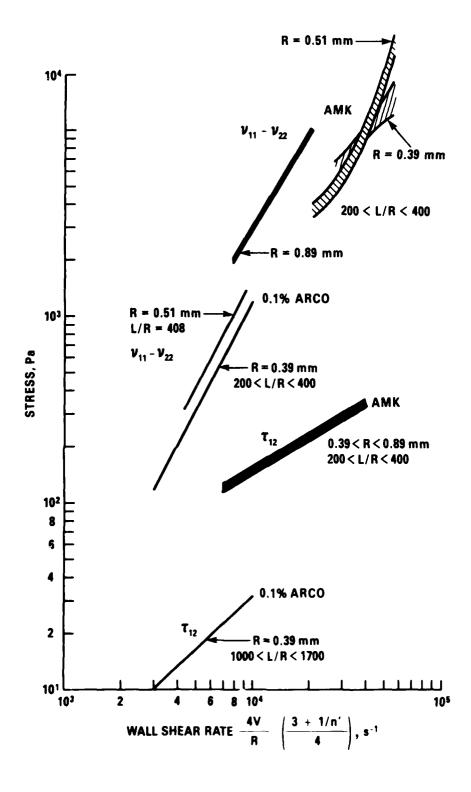


FIGURE 8. COMPARISON OF SHEAR AND NORMAL STRESSES OF TWO DIFFERENT ANTIMISTING FUELS

separated by a distance (H). The upper plate is free to move vertically under the influence of a constant load (F), while the lower plate is rotated at a constant angular velocity ($\boldsymbol{\Omega}$). If the sample contained between the two plates is a purely viscous liquid, then the gap (H) will decrease with time until the plates eventually come in contact. However, if the sample is a viscoelastic material, the gap may increase or decrease until the normal stresses just balance the load (F). Furthermore, by measuring the couple that prevents rotation of the upper plate, information can also be obtained regarding the shear viscosity. In order to make torsional balance experiments with a conventional Weissenberg Rheogoniometer (Model R18), it was necessary to make some modifications as suggested by Binding and Walters (reference 13). These changes are illustrated in figure 10 and involved: (1) replacing the torsional bar with a gap-measuring transducer that allows free movement of the air-bearing rotor; (2) attaching a metal frame to the air-bearing rotor; (3) connecting the metal frame to a balance pan and pulley arrangement so that the load on the upper plate can be varied; and (4) attaching a torque arm to the air-bearing rotor that rides against a load cell. The gap-measuring transducer was calibrated by bringing the two plates in contact and then measuring the separation produced by shims of known thickness. The torque arm (load cell) was calibrated with weights that were attached to string and suspended over a pulley.

In a typical experiment, the upper plate is brought in contact with the lower plate, and this point is referenced by a gap-setting transducer. Next, a shim is placed under the support, and weights are either added or removed from the balance pan to set the load on the upper plate. The plates are then separated by a screw adjustment, and a small amount of sample is poured into the dish that serves as the lower plate. Thus, in practice, the sample at the rim is surrounded by a "sea of fluid." The upper plate is then returned to the reference position by the screw adjustment and gap-setting transducer. Finally, the clutch to the motor that drives the lower plate is engaged and the shim under the support rod removed. The gap and torque are then measured as a function of time with a dual-pen recorder. It should be noted that in experiments with viscoelastic liquids, it is often not necessary to remove the shim in that the plates may separate spontaneously when the drive motor is started.

DISCUSSION OF RESULTS.

Newtonian Liquids. In order to check the accuracy and repeatability of the torsional balance rheometer, several non-rotational or squeeze-film experiments were conducted with liquids of known viscosities that were produced by blending Jet A with a mineral oil. The plate velocities (V) presented in figure 11 were obtained by graphical differentiation of the displacement-time curves obtained with a strip-chart recorder. The four different symbols for the lighter load $(6.2 \times 10^4 \text{ dynes})$ in figure 11 illustrate the typical experimental scatter that was observed for several runs with a Newtonian liquid. Considering the potential error that might be attributed to differentiation of the data, the repeatability illustrated in figure 11 is quite good. Nevertheless, these results show that the observed velocities were significantly higher than the

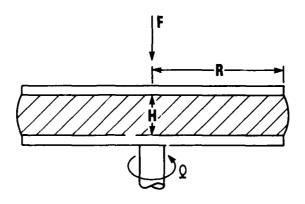


FIGURE 9. PRINCIPLE OF TORSIONAL BALANCE

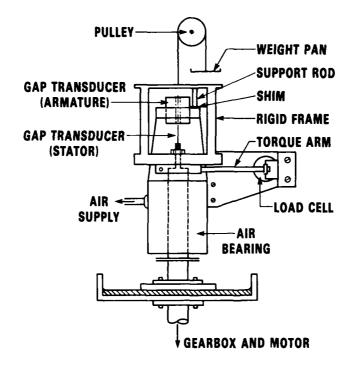


FIGURE 10. SCHEMATIC OF TORSIONAL BALANCE RHEOMETER

velocities predicted by the Stephan equation (solid lines in figure 11) for these experimental conditions:

$$V = \frac{2FH^3}{3\pi R^4 \mu} \tag{6}$$

However, the addition of 8 μm to the measured gap resulted in almost perfect agreement between the observed velocities shown and the Stephan equation. The experimental results reported by Brindley and co-workers (reference 14) showed excellent agreement with the Stephan equation without correcting the gap. However, their experiments were conducted with high viscosity (141 Pa·s) liquids at much larger gaps (300 to 800 μm), in which case an error of 8 μm would be difficult to detect.

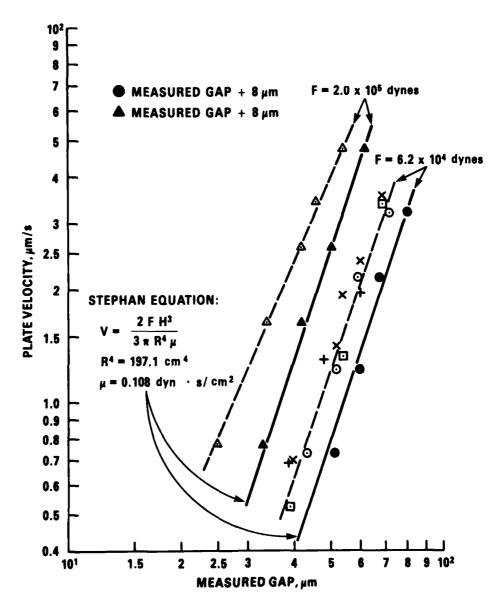


FIGURE 11. COMPARISON OF MEASURED PLATE VELOCITY WITH STEPHAN EQUATION

An independent check of the gap measurement was provided by rotational experiments in which the torque was measured as a function of time as the two plates approached each other. The gaps corresponding to these torque measurements were evaluated by:

$$H = \pi R^4 \mu/2T \tag{7}$$

and compared to the measured gaps in figure 12. Equation 7 is a special case of Equation 9 for n_T = 1. These results show that the measured gap is consistently off by $+\tilde{\mathbf{5}}$ μm . This observed difference between the measured and calculated (from torque) gaps usually varied from one experiment to the next, but was typically close to +7 µm. It should be noted that the maximum eccentricity between the two plates was approximately +5 μm. This value was measured by placing the two plates in contact and measuring the maximum deviation of the gap transducer when the lower plate was slowly rotated. Consequently, the average error of +7 µm in the measured gap is most likely due to misalignment Because of the agreement between these two independent of the plates. experiments with Newtonian liquids, a correction of +7 µm was added to the measured gap in calculating the shear rate at the rim of the plate for the experiments with antimisting fuels:

$$\dot{\gamma}_{R} = R\Omega/H \tag{8}$$

This correction was relatively minor in these experiments; however, it could be a limiting factor if very high shear rates $(10^5 \ \text{sec}^{-1})$ are required.

Polymer Solutions. Torsional balance experiments were conducted with AMK and 0.1 percent ARCO in Jet A. Typical behavior for the ARCO polymer is presented in figure 13. In contrast to the behavior of a purely viscous liquid in which the gap decreases with time after removal of the shim, with 0.1 percent ARCO in Jet A the gap increased with time until it reached an equilibrium value (1700 sec⁻¹) where the normal forces just balanced the imposed load of 8.6 x 10^4 dynes. The factor, F' = $3\pi\rho\Omega^2R^4$ ÷ 40, where ρ is the density of the fluid, was added to the load to correct for fluid inertia (reference 13).

The behavior of AMK under the same loading and initial shear rate is shown in figure 14. From these results it is evident that AMK reacts differently to the sudden application of shear than does the ARCO polymer. Many of these differences can be explained in terms of the critical shear rate and thixotropic nature of AMK. For example, the initial shear rate of 3800 sec⁻¹ is much higher than the critical shear rate of 1000 sec⁻¹ for AMK; consequently, there is rapid development of normal forces as soon as the clutch is engaged that causes the plate to separate. However, as the plates separate, the shear rate falls below the critical value (see 700 sec⁻¹ in figure 14). At this point, the normal forces are no longer generated, and the gap begins to decrease with time. As the gap decreases, the shear rate increases and eventually exceeds the critical shear rate (see 1000 sec⁻¹ in figure 14). At this point, normal forces are again generated, but they are not large enough to support the load. Consequently, after a momentary halt, the upper plate again falls (probably

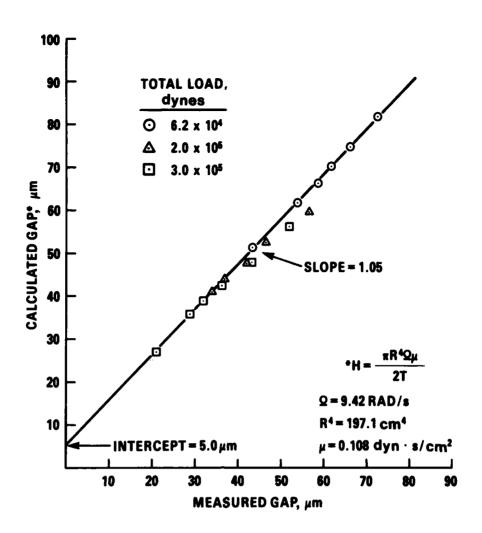


FIGURE 12. COMPARISON OF CALCULATED GAP FOR NEWTONIAN FLUID WITH MEASURED GAP

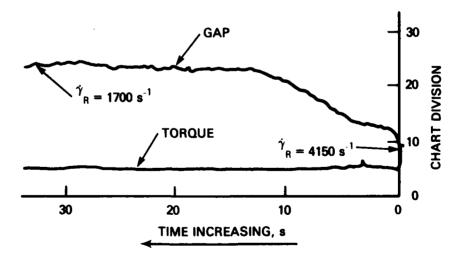
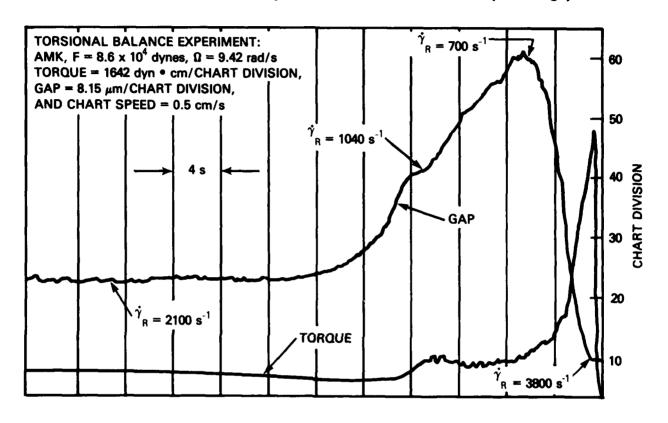


FIGURE 13. TORSIONAL-BALANCE MEASUREMENTS OF 0.1 PERCENT ARCO IN JET A $(F = 8.6 \times 10^4 \ dynes, \ \Omega = 9.42 \ rad/s)$

due to thixotrophy) until it reaches its equilibrium, at which the load is balanced by the normal forces. This transient behavior of AMK contains important information that should relate to its antimisting effectiveness and also should help to explain some of the anomalies that have been reported for AMK with rotational rheometers. However, only equilibrium measurements can be used to calculate normal stresses; therefore, for the purpose of this report, our attention will be focused on the equilibrium values of the torque and gap.



TIME INCREASING, s

FIGURE 14. TORSIONAL-BALANCE MEASUREMENTS OF AMK

Equations 9 and 10 (reference 13) were used to calculate the shear viscosity and normal stresses from the equilibrium torques and gaps measured at several different loadings and rotation speeds:

$$n_a = \frac{3T(1+n_T/3)}{2\pi R^3 \mathring{\gamma}_R}$$
, $n_T = d \log T/d \log \mathring{\gamma}_R$ (9)

$$v_1 - v_2 \Big|_{R} = \frac{2F(1+n_F/2)}{\pi R^2}$$
, $n_F = d \log F/d \log \hat{\gamma}_R$ (10)

The factor n_T in Equation 9 and n_F in Equation 10 are analogous to n' for capillary tube flow. The data for 0.1 percent ARCO (figure 15) show some scatter;

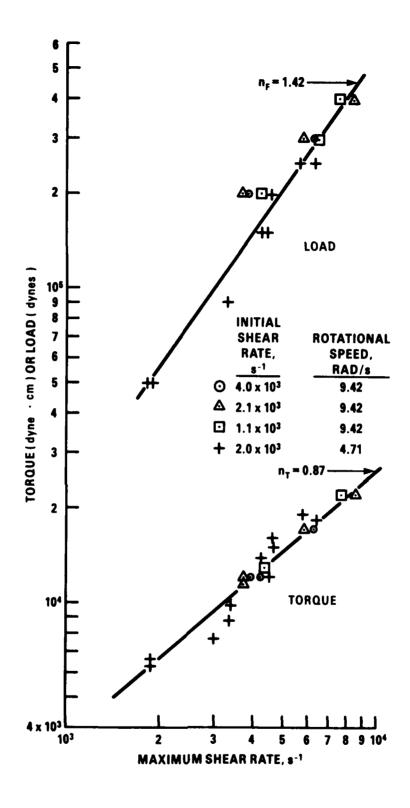


FIGURE 15. EQUILIBRIUM TORSIONAL-BALANCE MEASUREMENTS for 0.1 PERCENT ARCO IN JET A

however, at constant load the equilibrium torque and shear rate are essentially independent of the initial shear rate and rotational speed. Furthermore, at constant load the equilibrium shear rate and torque are also independent of the rotational speed. This invariance of the equilibrium shear rate and torque is an important check on the internal consistency of these measurements.

The results of similar experiments with AMK are presented in figure 16. Again, at constant load there was no measurable effect of initial shear rate. This in itself is rather surprising, considering the complex rheology of AMK in which there is a simultaneous buildup and breakdown of the shear-induced structure with time. Unfortunately, the results in figure 16 indicate anomalous effect of rotational speed for AMK that was not evident with the drag-reducing

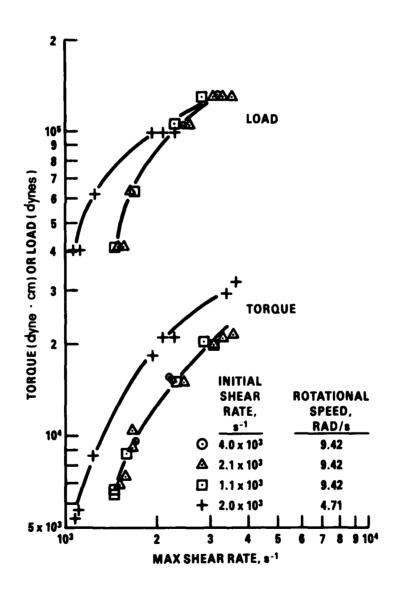


FIGURE 16. EQUILIBRIUM TORSIONAL-BALANCE MEASUREMENTS FOR AMK

In particular, both the torque and load curves were significantly higher at the lower (4.71 rad/s) rotational speed. Since the lower rotational speed is associated with smaller gaps, the higher torques cannot be attributed to the effects of wall-slip that are often observed with gel-like materials. Furthermore, the increased load with the smaller gaps is opposite to the increased load with larger gaps that was reported by Keentok and Tanner (reference 15) for experiments with a conventional parallel plate rheometer. The reason for this anomaly is probably associated with flow instabilities that have been observed for AMK with rotational viscometers (reference 2). It is important to mention that quasi-static data (i.e., $0 \le t \le 2$ sec in figure 14) indicated that the apparent viscosity of AMK was only slightly lower than the value measured for short (200 < L/R < 400) capillary tubes. In particular, the maximum (instantaneous) viscosity was 25 mPa·s at 2000 sec^{-1} , while the equilibrium (t > 24 sec in figure 14) viscosity at 2000 sec⁻¹ was only 8 mPa·s. This result suggests that thixotropic changes in structure and anomalies associated with the flow of AMK at high shear rates may be avoided in transient experiments if the time scale of the experiment is of the order of 1 second or less.

The dependency on rotational speed that was observed for AMK makes a comparison of these results with normal stresses calculated from die-swell of questionable value. However, a comparison of the shear and normal stresses for the ARCO polymer is shown in figure 17. The results in figure 17 show that the viscosity, based on capillary tube data, was slightly lower than torsional balance measurements. This is most likely the result of the large end corrections that had to be applied even with the very long tubes. While this small difference in viscosity is not very important, the large differences in the normal stresses are a matter of concern. In particular, the normal stresses measured by torsional balance were three to four times higher than normal stresses calculated from die-swell. It is important to note that that is exactly opposite to the results reported by Walters (reference 16) for polyisobutylene in decalin.

PHASE I CONCLUSIONS.

<u>Die-Swell Experiments.</u> Conclusions concerning die-swell experiments are:

1. AMK made with the improved FM-9 polymer behaves similarly to AMK made with the original FM-9 (i.e., it shear thickens at a critical shear rate, reaches a maximum apparent viscosity and then shear thins at higher shear rates); however, there are quantitative differences in which the improved FM-9 polymer results in a lower critical shear rate (1000 sec $^{-1}$ compared to 3000 sec $^{-1}$), and a higher apparent viscosity. AMK made with the improved FM-9 also appears to be more viscoelastic in that $\Delta P_{\rm e}$ is much larger.

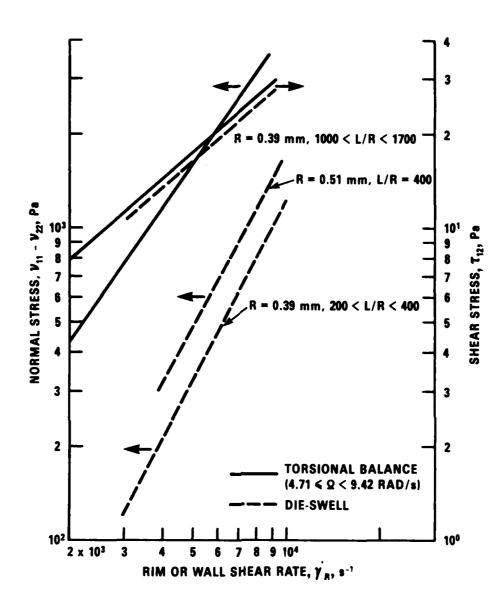


FIGURE 17. COMPARISON OF SHEAR AND NORMAL STRESSES FOR 0.1 PERCENT ARCO IN JET A CALCULATED FROM TORSIONAL-BALANCE AND DIE-SWELL MEASUREMENTS

2. At high shear rates ($\mathring{\gamma}_R = 10,000 \text{ sec}^{-1}$), the most outstanding rheological characteristics of both AMK and 0.1 percent ARCO are large normal stresses that are much higher than shear stresses.

Torsional Balance Experiments. Conclusions concerning the torsional balance experiments are:

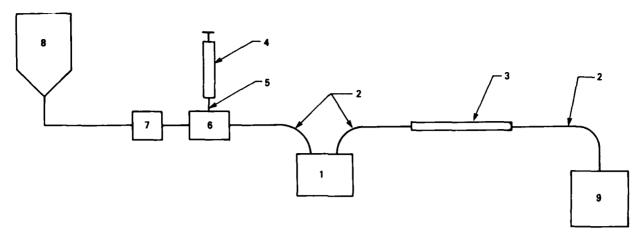
- l. The torsional balance rheometer is capable of measuring the shear and normal stress functions of dilute polymer solutions (0.1 percent ARCO) at high ($\mathring{\gamma}_R = 10,000 \text{ sec}^{-1}$) shear rates.
- 2. The thixotropic nature of the flow induced structure formed by AMK near $1000~{\rm sec}^{-1}$ makes the torsional balance method unsuitable for measuring normal stresses associated with the undisturbed structure. Unfortunately, an anomalous dependency on rotational speed also makes the equilibrium or stationary gap measurements for AMK of questionable value.
- 3. Quasi-static data indicate that the shear viscosity of AMK at 2000 sec $^{-1}$ (measured in a time frame of one to two seconds) is approximately the same as the maximum viscosity measured with short (200 < L/R < 400) capillary tubes. This result suggests that difficulties associated with thixotrophy and flow instabilities can be avoided with AMK if shear and normal stresses are measured in transient step-strain or step-shear rate experiments.
- 4. Depending on the particular polymer solution, normal stresses calculated from die-swell measurements can be larger or smaller than normal stresses calculated from torsional balance data.

PHASE II--RHEOLOGICAL PROPERTIES OF AMK

DIE-SWELL RHEOMETER.

Description of Experiment. Experiments dealing with the effects of polymer concentration and method of blending AMK (batch and in-line) on the shear and normal stress functions were conducted in Phase II. In addition to the video-micrometer used in Phase I, an optical "die-swell tester" (Manufactured by C.W. Brabender Instruments Inc.) that automatically measures an average jet diameter was also evaluated. A second modification to the die-swell rheometer (reference 17), eliminated the need for measuring viscoelastic end corrections $(\Delta\,P_e)$. This improved viscometer design utilizes tee connectors that have the same ID as the capillary tube for pressure taps. While the total pressure at any tap would be in error due to the "hole pressure" effect, by measuring pressure differences these errors would be expected to cancel.

In-line blending of laboratory quantities of AMK from Avgard and Jet A was accomplished by placing a weighed amount of Jet A in the fuel reservoir (see figure 18 for schematic of the laboratory in-line blender). To ensure uniformity of the Avgard slurry, it was thoroughly stirred just before a sample



NOTE:

TUBE LENGTH 6-3 20"

TUBE LENGTH 3-9 15"

TUBE LENGTH 8-7-6 NO SPEC.

TUBE SIZE 8-7-6 3/8"

COMPONENTS

- 1. PUMP
- 2. TYGON TUBING
- 3. STATIC MIXING TUBE
- 4. SYRINGE
- 5. COUPLING
- 6. SYRINGE MOUNT
- 7. BALL VALVE
- 8. SUPPLY CONTAINER
- 9. CATCH CONTAINER

FIGURE 18. ANTIMISTING FUEL LABORATORY BLENDER

was taken. The amount of slurry needed to produce the desired polymer concentration (typically 34 gm of slurry in 2800 gm of Jet A resulted in 0.3 percent FM-9) was weighed into a syringe which was then attached to the syringe mount. The pump (1.0 L/min delivery rate) was then switched on, and the rate of slurry injection was controlled manually so that a small amount of fuel would be left in the reservoir to flush the slurry from the lines when the syringe was empty. The resulting dispersion of slurry and Jet A passed through a 0.25-in. static mixing tube and was caught in a glass beaker. The rate of clarification of this dispersion was monitored with a nephlometer.

DISCUSSION OF RESULTS.

Apparent Viscosity. Since the majority of the experiments in Phase I were made at high shear rates ($\mathring{\gamma}_R > 10,000 \text{ sec}^{-1}$), additional experiments were conducted in Phase II at shear rates nearer to the critical. The data in figures 19 and 20 are the measured pressures (using the original die-swell rheometer shown in figure 1) with batch-blended AMK for different values of 4V/R and L/R (R = 1.47 mm). These data show that ΔP_e starts to become important for $800 < 4\text{V/R} < 1000 \text{ sec}^{-1}$, and then increases monotonically for $4\text{V/R} > 1000 \text{ sec}^{-1}$. It should be noted that ΔP_e in figure 19 begins to be a significant factor at the value of 4V/R associated with the sudden increase in the effective viscosity (i.e., the critical shear rate) in figure 21. More importantly, after the data in figure 21 are corrected for ΔP_e , they no longer show a dependence on L. Furthermore, although the end corrections for R = 1.47 mm

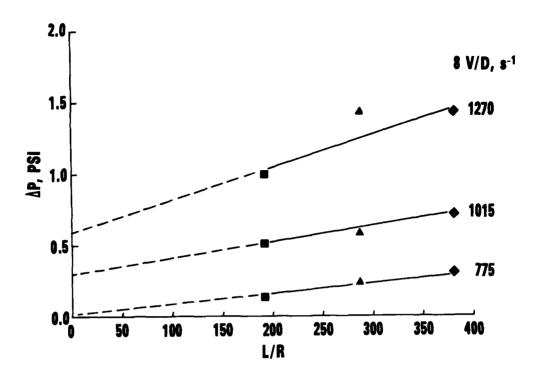


FIGURE 19. ESTIMATED $^{\triangle}P_{e}$ FOR AMK NEAR THE CRITICAL SHEAR RATE (R = 1.47 mm)

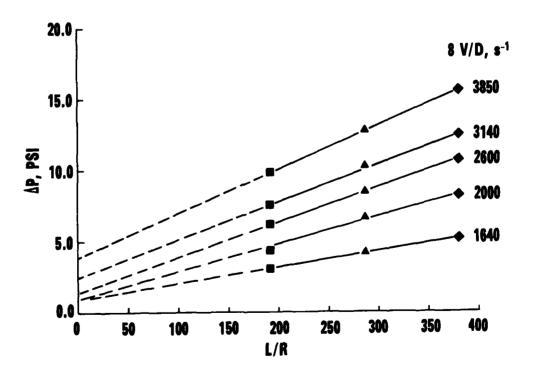


FIGURE 20. ESTIMATED ΔP_{e} FOR AMK ABOVE THE CRITICAL SHEAR RATE (R = 1.47 mm)

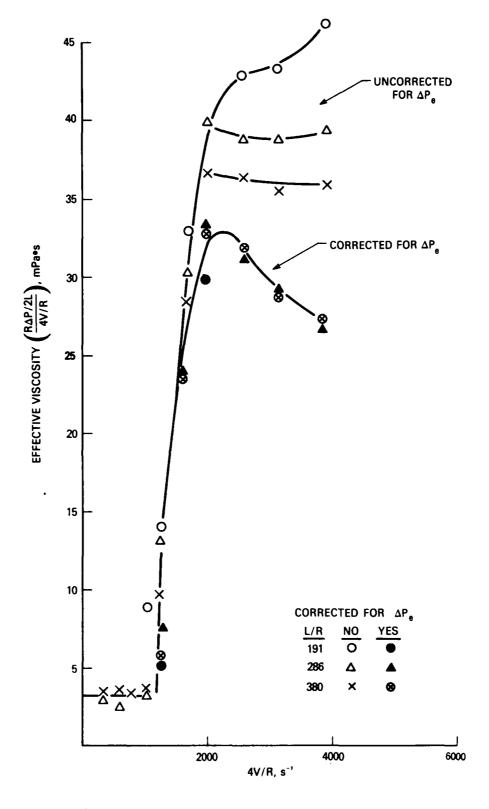


FIGURE 21. INFLUENCE OF END CORRECTIONS ON THE EFFECTIVE VISCOSITY OF AMK (R = $1.47\ mm$)

are much smaller than for R=0.51 mm (figure 22), after these corrections have been made, the effective viscosities for these different size tubes show essentially the same dependence on 4V/R. In particular, the maximum viscosities are 33 mPa·s and 30 mPa·s for R=1.47 mm and 0.51 mm, respectively. These values are reasonably close to the 36 mPa·s that was calculated for the data in figure 4.

Although these experiments have shown that end corrections result in apparent viscosities that are independent of R and L, the procedure of estimating ΔP_{e} from measurements with at least three different length tubes is too tedious for a real-time quality-control test. Consequently, experiments were conducted with a capillary viscometer that measures differential pressures and therefore does not require end corrections (reference 17). Furthermore, by measuring the differential pressure at different positions along the tube, the effect of residence time on viscosity was determined. The results of these experiments for several different in-line blended samples of AMK are presented in figure 23. The residence times in the tubes at the shear rate that produced the maximum effective viscosity (2000 sec^{-1}) was 1.0 and 3.4 seconds at stations 1-2 and 3-4, respectively (i.e., $t_{1-2} = 4(500)/2000 \text{ sec}^{-1} = 1.0 \text{ sec}$ and $t_{3-4} = 4(1700)/2000 \text{ sec}^{-1} = 3.4 \text{ sec}$). Consequently, the higher maximum viscosity at station 1-2 (45 mPa·s compared to 35.5 mPa·s) indicates that a residence time of the order of 3.4 seconds causes a significant breakdown in the structure of AMK near the critical shear rate. This result is in good agreement with the results for short (200 < L/R < 400) and long (L/R = 1660) tubes shown in figure 4. While the critical shear rate in figure 23 is very close to the value observed for experiments in which end corrections were required (approximately 1000 sec^{-1}), the maximum apparent viscosity for station 1-2 in figure 23 is significantly higher than the maximum viscosities that were observed earlier (45 mPa·s compared to 30-36 mPa·s). Since these earlier experiments were made with batch-blended AMK, while the results in figure 23 are for AMK made with the Avgard slurry (in-line blending), the different values for the maximum viscosity may be caused by the different methods of blending. However, batch-to-batch differences in the polymer, or overestimation of $^{\triangle}P_{P}$ may also be responsible. Matthys (reference 18) reported capillary tube measurements (R = 0.5 mm, L/R = 1174) for in-line blended AMK (age--1 day) in which the maximum apparent viscosity was only 17 mPa·s. It is important to note that Matthys used long tubes to minimize end corrections. Consequently, this low viscosity can be partly explained by thixotropic changes that occur with long tubes (figure 23). Nevertheless, the critical shear rate of 3000 sec-1, reported by Matthys, is uncharacteristic of the improved FM-9 polymer unless it has been degraded.

The concept of measuring the maximum apparent viscosity without the need of is particularly important for corrections for ΔP_{c} quality-control of AMK. Furthermore, the results in figure 24 show that the maximum apparent viscosities that were measured without making end corrections for nine different samples blended from Avgard (three each at 0.25, 0.30 and 0.35 percent) exhibit a strong linear dependency on polymer concentration. It is important to note that the results presented in figure 24 for in-line blending of AMK in the laboratory are in excellent agreement with the sample taken from the No. 3 tank and tested approximately 20 hours after 12,000 gallons of AMK had been blended for the Controlled Impact Demonstration.

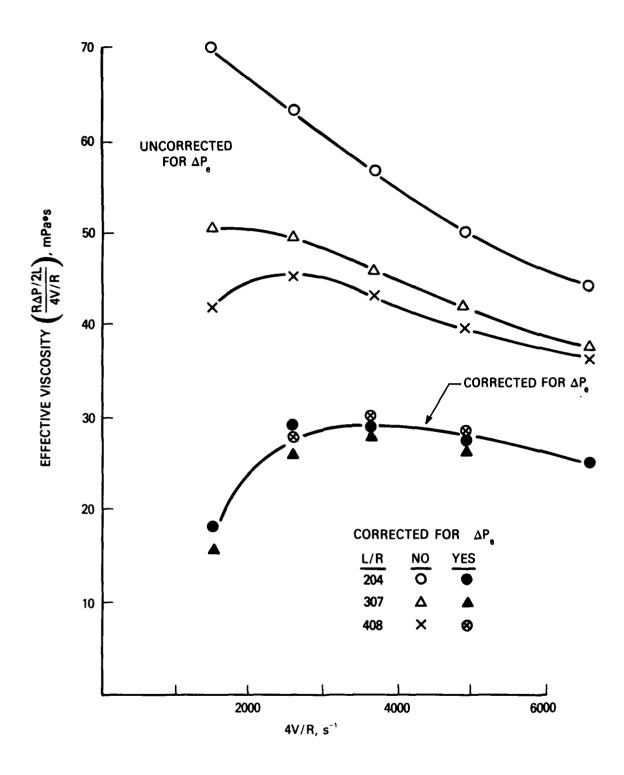


FIGURE 22. INFLUENCE OF END CORRECTIONS ON THE EFFECTIVE VISCOSITY OF AMK (R = 0.51 mm)

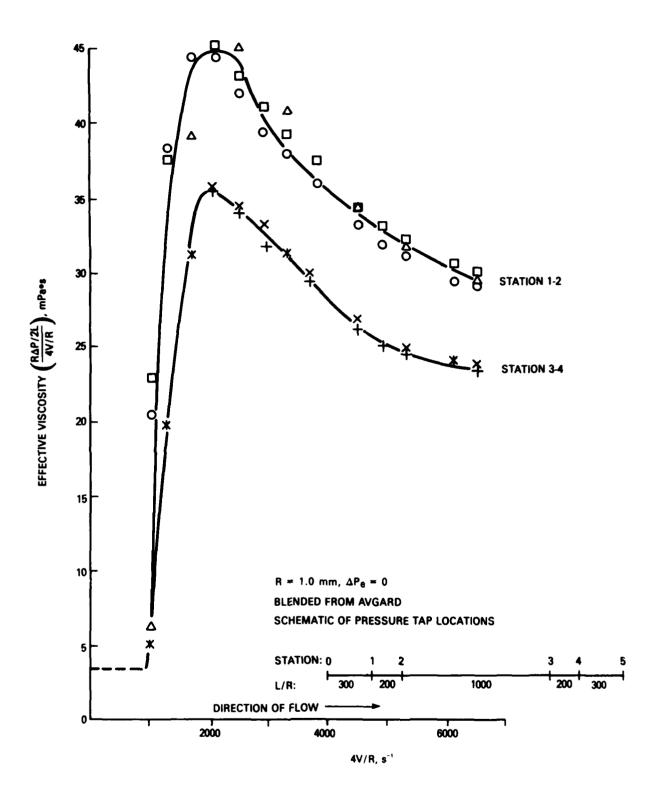


FIGURE 23. EFFECT OF RESIDENCE TIME ON VISCOSITY OF AMK

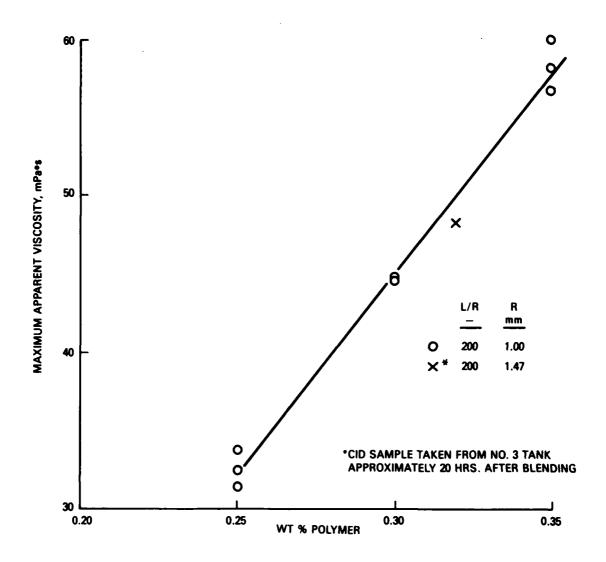


FIGURE 24. EFFECT OF POLYMER CONCENTRATION ON THE MAXIMUM APPARENT VISCOSITY OF AMK ($\Delta P_e = 0$)

that similar normal stresses were Die-Swell. Earlier it shown was calculated from die-swell measurements for AMK and 0.1 percent ARCO in Jet A (figure 8). This finding is important in that both of these fuels have been shown to provide similar mist-fire protection in fuel-spillage experiments conducted by the FAA. However, in order for die-swell measurements to be useful for quality control, it is necessary for these measurements to detect changes polymer concentration that significantly change mist-fire protection. Consequently, experiments were conducted in which the concentration of FM-9 was varied between 0.25 and 0.35 wt percent, while the ratios of the carrier fluid components to the polymer were held constant. The results of these experiments are shown in figures 25-27.

In addition to die-swell measurements for AMK, figure 25 contains data for a Newtonian liquid (Jet A/Mineral Oil). The limiting die-swell ratio for $R_e > 100$ of 0.93 is slightly higher than the theoretical value of 0.87 (Eq. 5, n=n'=1.0). The reason for this higher value can not be explained in terms of normal stresses for Newtonian liquids. However, it has been observed by others, and it is in good agreement with the measurements that were discussed earlier (see R=0.635 mm in figure 7).

As expected, the die-swell ratios for AMK at high shear rates that are shown in figure 25 are significantly larger than a purely viscous liquid (i.e., AMK is viscoelastic at high shear rates), and are an increasing function of concentration. However, the sensitivity of die-swell to polymer concentration is not as good as expected. Even after normal stresses were calculated from these die-swell ratios and the measured shear dependencies for 7000 < 4V/R $< 18,000 \text{ sec}^{-1}$ (n = n' = 0.74, 0.67 and 0.58, for 0.25, 0.30 and 0.35 percent FM-9, respectively) using Equation 5, the changes in normal stresses with polymer concentration are still relatively small (figure 26). Furthermore, when these normal stresses are expressed in terms of the wall shear rate by Equations 1 and 2, these small differences in the normal stresses are no longer evident (figure 27). The fact that the normal stress function for AMK in figure 27 does not increase with concentration conflicts with the large difference in antimisting protection that is provided by 0.25 and 0.35 wt percent FM-9. The reason for this discrepancy has not been proven. However, when these findings were presented at the IX International Congress on Rheology, it was the opinion of several of the participants that Equation 5 is based on assumptions that are not generally valid for viscoelastic material (see reference 19 for a critique of Eq. 5), and that a numerical (finite element) simulation method would be a better approach for relating die-swell to the rheological properties of a polymer solution (references 20 through 22).

Since the normal stresses calculated from die-swell ratios do not show the proper dependency on polymer concentration, they are of little value for quality Nevertheless, die-swell is a viscoelastic phenomenon and should be a valuable criterion for quality control. Consequently, emphasis was placed on improving the sensitivity of these measurements to polymer content by conducting experiments at lower shear rates (i.e., shear rates nearer to the critical). The reason for the expectation of larger die-swell ratios at lower shear rates was that very large ratios $(d_i/D = 3.7 \text{ at } 4V/R = 4800 \text{ sec}^{-1})$ were observed for AMK during the early development of the die-swell rheometer (reference 23). Consequently, experiments were conducted in which the die-swell ratios of batch-blended AMK were measured with the optical "die-swell tester" as a function of 4V/R. Two different size tubes (R = 0.89 mm and 1.47 mm) were used so that 4V/R could be varied while maintaining $N_{Re} > 100$. The primary reason for keeping $N_{\mbox{\scriptsize Re}}$ > 100 is that this is a requirement for calculating normal stresses from die-swell with Equation 5; however, it is now realized that high values of NRe suppress die-swell.

The data in figure 28 show that the measured die-swell for a Newtonian liquid at $N_{\mbox{\scriptsize Re}}>100$ and $4\mbox{\scriptsize V/R}>8$ x 10^3 sec $^{-1}$ is very close to the theoretical value of 0.87 calculated from Equation 5. It should be noted that the assumptions for Equation 5 are generally valid for Newtonian liquids, but not for highly elastic materials. The reason for the closer agreement obtained with the optical

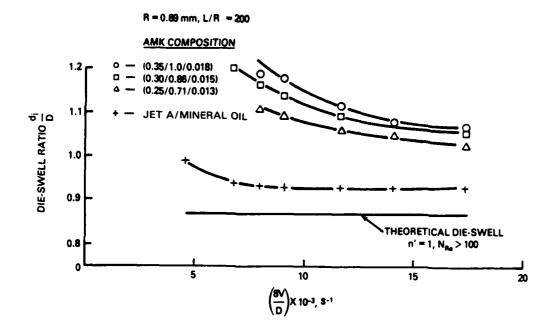


FIGURE 25. EFFECT OF AMK COMPOSITION ON DIE-SWELL RATIO

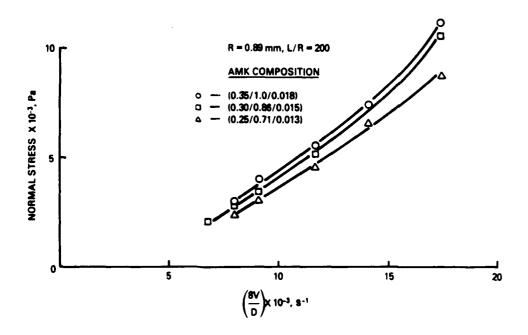


FIGURE 26. EFFECT OF AMK COMPOSITION ON NORMAL STRESS BEHAVIOR

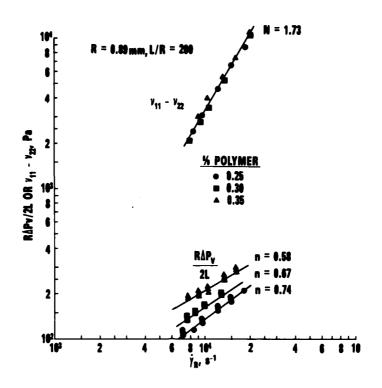


FIGURE 27. EFFECT OF POLYMER CONCENTRATION ON SHEAR AND NORMAL STRESSES

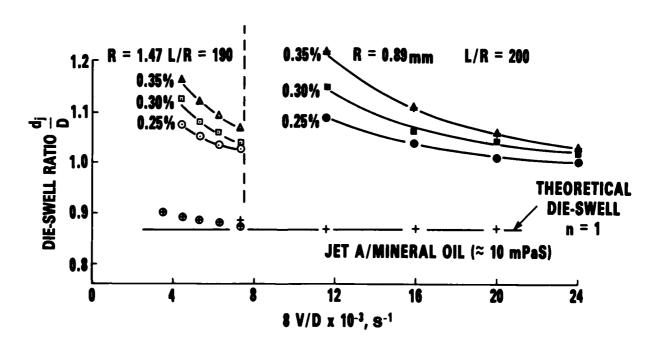


FIGURE 28. EFFECT OF AMK COMPOSITION, APPARENT SHEAR RATE, AND TUBE DIAMETER ON DIE-SWELL MEASUREMENTS $\rm N_{Re} > 100$ (Optical Die-Swell Tester)

die-swell tester rather than with the video-micrometer (0.93 in figure 25) is probably associated with the different ways in which these devices measured di. For example, in order to determine di with the video micrometer, the operator must make a subjective decision regarding the boundaries of the jet (which are usually not stationary) and the relative positions of cursor lines. On the other hand, an average value of di (over some axial distance) is obtained automatically with the die-swell tester. Nevertheless, the differences in 0.93 and 0.87 determined by these two methods are not very large. Furthermore, die-swell ratios for AMK (0.3 percent with R = 0.89 mm) are essentially the same for these two methods (see figures 25 and 28). More importantly, instead of the large die-swell ratio of 3.7 at $4V/R = 4800 \text{ sec}^{-1}$ that had been observed earlier for AMK with R = 0.5 mm, the die-swell ratio was only 1.1 at this same value of 4V/R but with R = 1.47 mm (figure 28). Before discussing the reason for this result, the data in figure 29 will be considered first. These data show that it takes approximately 30 minutes for die-swell differences in in-line blended AMK to become insignificant. This finding is in good agreement with flammability tests that indicate approximately 30 minutes are required for AMK to develop its full potential as an antimisting fuel.

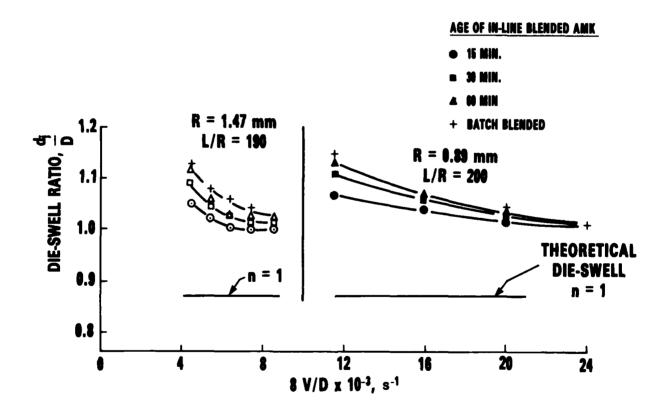


FIGURE 29. EFFECT OF SAMPLE AGE, APPARENT SHEAR RATE, AND TUBE SIZE ON DIE-SWELL MEASUREMENTS (0.3 PERCENT FM-9) $N_{\mbox{Re}} > 100$

The general tendency for die-swell to decrease with increasing 4V/R and R fixed can be attributed to the fact that fluid inertia reduces viscoelastic effects. Therefore, the large die-swell ratios that were observed during the initial development of the die-swell rheometer were probably a result of the low inertial effects (i.e., low N_{Re}) associated with relatively small (R = 0.5 mm) tubes. For example, at constant 4V/R (or 8V/D), N_{Re} is 1/9th as large for R = 0.5 mm as for R = 1.47 mm [i.e., N^{\prime}_{Re} = DVp/ n_a = (D²/8)(8V/D)(p/ n_e)]. Consequently, much larger effects of concentration on die-swell would be expected if experiments were conducted with relatively small tubes and at shear rates close to the maximum apparent viscosity, thus keeping N_{Re} as small as possible.

REAL-TIME DEGRADATION/FILTRATION.

Introduction. Despite the fact that AMK can form a gel-like structure, it is possible for AMK to flow through a filter if the local deformation rate is below a critical value. Since flow through a typical filter is generally too complex to calculate the local deformation rate, the most that can be said is that filter plugging can be prevented provided that the superficial velocity (V = Q/A, where Q is the flow rate and A is the flow area) is less than acritical value. Thus the critical velocity (Vc) has been defined as the highest velocity at which AMK can flow through a filter without causing the pressure to increase with time (references 5, 6, 24). Since the onset of filter plugging is related to the critical deformation rate, $V_{\mbox{\scriptsize c}}$ generally depends on filter type (surface or depth) and geometry (such as pore size, shape, tortuosity, etc.). For conventional aircraft filters, the critical velocity for AMK is so low that very large areas would be required. However, reducing the molecular weight of the polymer by mechanical degradation increases the critical velocity to a level that will allow AMK to flow at the high flow rates associated with take-off and landing.

Description of Experiments. It was originally intended to develop a test that could continuously measure the filtration characteristics of AMK while it was being degraded; however, the low lubricity of Jet A and AMK resulted in rapid pump wear even at modest pressures of 1000 to 2000 psi. Since a small hydraulic pump with specially hardened surfaces such as the one used by Mannheimer (references 5 and 25) was not commercially available, it was necessary to resort to a batch degradation/filtration experiment.

The essential components of the apparatus that was used in these simultaneous degradation/filtration experiments are shown in figure 30. In a typical experiment, approximately 1200 mL of AMK is charged to the rod-side of a hydraulic cylinder by applying nitrogen pressure to the fuel reservoir. After the cylinder is loaded with fuel, the hydraulic pump is started, and the displacement of AMK is controlled by a variable-speed-drive-motor. AMK is degraded by maintaining a large pressure drop ($\Delta P_{\rm max}$ = 5000 psi) across a 0.25-inch needle valve. Immediately after the needle valve, the AMK flows through a small section of metal screen or paper filter that is held between two metal backup plates in a 0.5-inch stainless steel union (see reference 6 for details of filter holder). The area of the filter was varied by changing the size of the holes in the backup plates. The pressure drop across the filter was measured as a function of time and changes in ΔP of less than 0.01 psi could easily be detected.

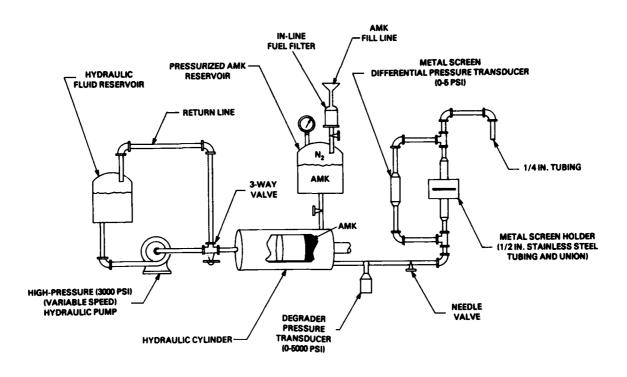


FIGURE 30. SIMULTANEOUS DEGRADATION/FILTRATION APPARATUS

Aircraft fuel filters usually have large areas (3000 to 4000 cm²) so that the superficial velocity even at take-off is low (V < 1 cm/s). However, fuel delivery components such as the fuel controller are protected by screens with much smaller areas (10 cm²) that result in high velocities (10 < V < 75 cm/s). In order to obtain these high velocities and sustain relatively long flow times with the limited (1200 mL) amount of fuel in the hydraulic cylinder, the use of very small filter areas $(0.04 < 4 < 0.2 \text{ cm}^2)$ was necessary. Since these filters would be quickly plugged by lint or dirt, the fuel was passed through an in-line filter (Gasoline filter-1-GF451, AC Spark Plug Division) before it entered the reservoir (see figure 30). In spite of this precaution, debris and wear particles that appeared to originate from the 0-rings on the hydraulic piston resulted in partial plugging of screens even with Jet A. This problem was essentially eliminated by placing a second filter element (not shown in figure 30) immediately after the needle valve. The area of this filter was large enough (200 cm²) that undegraded AMK could flow through it at the maximum flow rate (167 mL/min) of the hydraulic pump without difficulty.

DISCUSSION OF RESULTS.

Square-Mesh Metal Screens. Figure 31 shows the results of simultaneous degradation/filtration experiments for in-line blended AMK with square mesh metal screens. The fuel used in these experiments was degraded 30 minutes

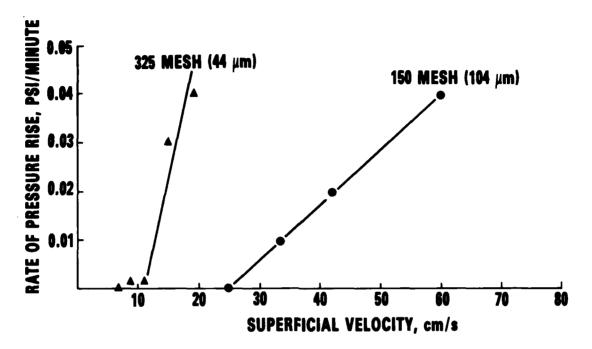


FIGURE 31. EFFECT OF SUPERFICIAL VELOCITY ON RATE OF PRESSURE RISE

(In-Line Blended AMK, Age--30 min, 27.6 kWs/L)

after blending and had the following physical properties just before it was degraded: FR = 35-36, OC = 2.2 mL/30 sec and Nephelometer = 10 to 11 NTU. Nephelometer values for in-line blended AMK decreased from 40 to 50 NTU after 5 minutes to 8 to 10 NTU after 30 minutes. Generally there was very little decrease in NTU after 30 minutes. The data in figure 31 clearly illustrate the critical velocity concept and also show that V_{C} generally is different with different size screens (i.e., $V_c = 11$ cm/s and 24 cm/s for the 325 mesh and 150 mesh screens, respectively). These results represent the first time that V_C has been measured while AMK was being degraded. This accomplishment is particularly important because $V_{\mbox{\scriptsize c}}$ for degraded AMK often decreases with time. Furthermore, this time-dependent behavior is even more evident with in-line blended AMK if it is degraded soon after blending. Another important aspect illustrated by the data in figure 31 is the relatively low rate of pressure rise of degraded AMK at velocities that are well above V_c (i.e., \leq 0.05 psi/min at $V = 2V_c$). In fact, there are some indications that the rate of plugging of AMK with square-mesh metal screens may eventually decrease and approach zero if the flow is sustained for a long enough time.

The data in figure 32 are the results of simultaneous degradation/filtration experiments with batch-blended AMK (FR = 65, OC = $1.8~\rm mL/30$) in which the specific degrader power was the same as with the in-line blended fuel shown in figure 31. Rheological differences between batch-blended AMK made with the newer FM-9 polymer are characterized by a higher FR (65 compared to 40) and a lower OC ($1.8~\rm to~2.0~mL/30~sec$ compared to $2.6~\rm to~2.8~mL/30~sec$). The

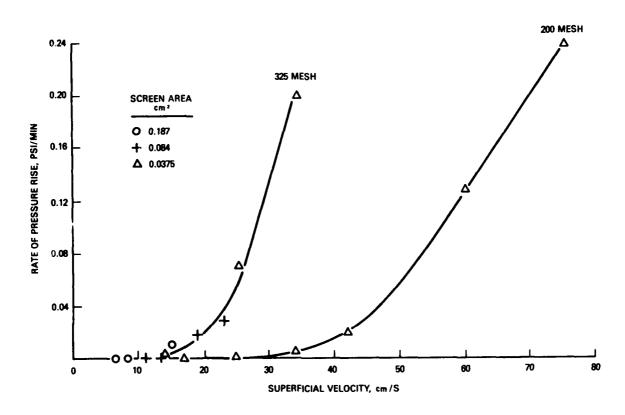


FIGURE 32. EFFECT OF SUPERFICIAL VELOCITY ON RATE OF PRESSURE RISE
(Batch-Blended AMK, 27.6 kWs/L)

results of these experiments show that $V_{\rm C}$ with the 325 mesh screen is only slightly higher (13 to 14 cm/sec) for batch-blended AMK than for in-line blended AMK (age--30 minutes) at the same degrader power. Furthermore, the rate of pressure rise at $V = 2V_{\rm C}$ is about the same (0.05 psi/min) as for in-line blended AMK after 30 minutes. This finding conflicts with the filtration ratios of these degraded fuels which indicated that the batch-blended fuel was more highly degraded. In order to minimize changes that might occur following degradation, these filtration ratios were measured while the samples were still warm (30°C). Nevertheless, the values of 1.12 and 10 for batch-blended and in-line blended AMK (age--30 minutes), respectively, illustrate why it was originally felt that in-line blended AMK would be difficult to degrade unless it was allowed to equilibrate for several hours. It is important to point out that the results

in figure 32 also show that $V_{\rm C}$ increases with pore size but is independent of the screen area (A). This latter result is the reason $V_{\rm C}$, rather than $Q_{\rm C}$, should be used to characterize filter plugging.

Because of the simple geometry of square-mesh screens, the pore size (D_p) and open area (A_0) can be calculated from the mesh count (M) and wire size (W). Furthermore, if these quantities are used to express V_c in terms of a critical deformation rate (V_c/D_pA_0) , the onset of filter plugging with different size screens occurs at approximately the same value. For in-line blended AMK that is degraded at a specific power of 27.6 kWs/L 30 minutes after blending, the critical deformation rate is 8 x $10^3~{\rm sec}^{-1};$ for batch-blended AMK, it is only slightly higher (1.1 x $10^4~{\rm sec}^{-1}).$ It is doubtful that the deformation rate is numerially equal to V_c/D_pA_0 , but the invariance of V_cD_p/A_0 that is illustrated in table 2 suggests that it is at least proportional to the deformation rate for a limited range of experimental conditions:

TABLE 2. CRITICAL DEFORMATION RATE FOR AMK WITH SQUARE-MESH SCREEN

				Degraded AMK (27.6 kWs/L)				
	Wire	re			In-Line Blending (Age30 min)		Batch-Blended	
Mesh Count	$\frac{\text{Size}}{\text{in.x10}^3}$	D _p *, _μm	<u>A₀*</u>	V _c ,	$\frac{v_c/D_pA_o}{\sec^{-1}}$	V _c ,	V _c /D _p A _o , sec 1	
70	6.5	200	0.30	51	8.5×10^{3}			
150	2.6	104	0.37	24	6.6×10^3			
200	2.1	74	0.34	21	$8.3x10^{3}$	25	1.1×10^4	
325	1.4	44	0.30	11	7.5×10^3	13.5	9.2×10^{3}	

The effect of specific degrader power on $V_{\rm C}$ and $V_{\rm C}/D_{\rm p}A_{\rm O}$ for a 200 x 200 squaremesh screen is illustrated in figure 33. These data indicate that there is a specific power threshold below which very little degradation occurs. For example, the critical velocity for undegraded AMK is 0.9 cm/s and it increases to between 1.5 and 2.5 cm/s at 6.7 kWs/L. However, from 6.7 to 13.4 kWs/L (1000 to 2000 psi) $V_{\rm C}$ increases to 13 cm/s, while at specific powers greater than 13.4 kWs/L, the increase of $V_{\rm C}$ is more gradual. Compared to the significant levels of degradation that can be achieved for some polymer solutions at less than 1 kWs/L (reference 26), the threshold level of 6.7 kWs/L for AMK indicates that the FM-9 polymer is relatively resistant to mechanical degradation. Because of the invariance of the critical deformation parameter with different size screens, the results presented in figure 33 in terms of $V_{\rm C}/D_{\rm p}A_{\rm O}$ should estimate the specific degrader power needed to prevent plugging of different fuel delivery components provided that these components contain square-mesh metal screens and the fuel temperature is near ambient.

 $[*]D_p = (1 - WM)/M, A_o = (D_pM)^2.$

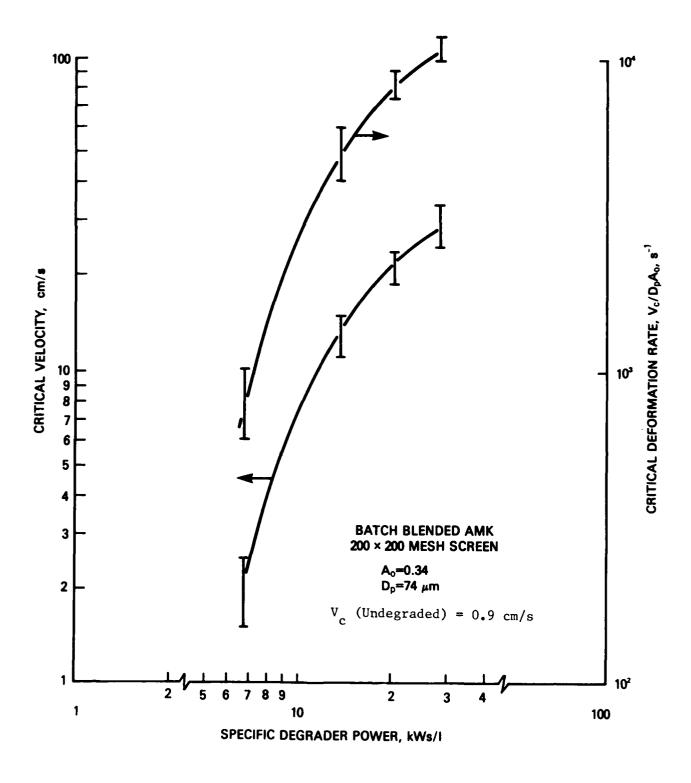


FIGURE 33. EFFECT OF SPECIFIC DEGRADER POWER ON CRITICAL VELOCITY AND CRITICAL DEFORMATION RATE

A few experiments were conducted to demonstrate that $V_{\mathbf{c}}$ decreases with time after degradation. In these experiments, in-line blended AMK was degraded at 27.6 kWs/L 30 minutes after blending and V_c was typically 11 cm/s for a 325 mesh screen. Approximately 18 hours later, the same degraded samples were retested; however, this time the needle valve was set fully open so that no further degradation would occur, and the critical velocity was only 2 cm/s. Furthermore, while no gel build-up was found after the degradation/filtration experiment, a heavy coating of gel was present on both the upstream and downstream sides of the screen after the degraded sample was retested. Similar experiments were conducted with in-line blended AMK that had equilibrated several days (5 to 7) before it was degraded. The value of V_C for these equilibrated samples was essentially the same as for the 30-minute samples (i.e., 11 cm/s for 27.6 kWs/L with a 325-mesh screen) but was only 2 cm/s the next day. The major difference between AMK degraded 30 minutes after blending and equilibrated AMK is that the rate of plugging with the latter was very low (0.02 psi/min at 4 cm/s) and no gel could be found on the screen.

<u>Paper Filters.</u> Simultaneous filtration/degradation experiments have been conducted with AMK using different filter paper materials. Unlike a square mesh metal screen, filter paper has a wide distribution of pore sizes and shapes. The nominal pore size (D_N) , which is calculated by displacing a liquid of known surface tension from the paper with air, attempts to characterize this complex structure with a single measurement. Nevertheless, the nominal pore size has been found to be useful in characterizing filter plugging by AMK with different paper material.

The porosity (ϵ) of filter paper which is roughly equivalent to the fraction of open area (A_0) for a screen is difficult to measure accurately because the thickness of the paper is typically only 0.018 to 0.020 inch. Nevertheless, based on the measured weight and dimensions of the paper, along with the reported bulk density of the fibers (A solid density of 0.96 gm/mL was estimated from the density of cellulose [0.90 gm/mL] and the binder [1.5 gm/mL] that was 10 percent by weight of the solid.), the porosities for 40- and 80-µm paper obtained from Facet were estimated to be 0.29 and 0.43. Using this same procedure and assuming the 40-µm paper from the main fuel filter for a JT8D engine contained the same amount of binder, the porosity was estimated to be 0.32. Thus, within experimental accuracy, the two 40-µm papers have the same porosity and the 80-µm paper has a significantly higher porosity.

The results in table 3 summarize the effects of specific degrader power, the type of blending procedure, and the age of the sample on the filtration properties of AMK at ambient temperature. The first series of experiments was conducted with batch-blended AMK. The critical velocity for the 80- μ m (Facet) paper at 27.6 kWs/L was estimated to be 1.15 to 1.20 cm/s for repeat runs. This is roughly 1/20 of the critical velocity (approximately 25 cm/s) for 74- μ m metal screens. This result illustrates the higher sensitivity of paper filters to plugging by AMK, and is probably caused by the more complex flow geometry of paper filters compared to metal screens.

TABLE 3. CRITICAL DEFORMATION RATE FOR AMK WITH PAPER FILTERS

				Filtration Pa	rameters
Pape	r Filters		Specific Degrader		
Source	D _N , μm	<u>ε</u>	Power, kWs/L	V _C , cm/s	$\frac{V_{C}/D_{N}\varepsilon}{s}$
Facet	80	0.43	20.7	0.55	160
Facet	40	0.29	20.7	0.28	240
JT8D Engine	40	0.32	20.7	0.30	230
					210
Facet	80	0.43	27.6	1.15	350
Facet	80	0.43	27.6	1.12	340
Facet	80	0.43	27.6	1.30	380
Facet	40	0.29	27.6	0.45	390
JT8D Engine	40	0.32	27.6	0.36	280
JT8D Engine	40	0.32	27.6	0.66	520_
					380
Facet	80	0.43	34.5	2.7	700
Facet	40	0.29	34.5	0.59	510
JT8D Engine	40	0.32	34.5	0.82	630
JT8D Engine	40	0.32	34.5	0.75	580
					600

Although the critical deformation rate parameter V_c/D_pA_0 for screens is much higher than $V_c/D_N\varepsilon$ for paper (i.e., $1 \times 10^4~{\rm sec}^{-1}$ compared to $3.5 \times 10^2~{\rm sec}^{-1}$ at $27.6~{\rm kWs/L}$), the use of this concept appears promising for normalizing data obtained with different paper filters. For example, at $27.6~{\rm kWs/L}$, the critical velocity changed with pore size but $V_c/D_N\varepsilon$ was essentially constant (see table 3). With the exception of the high (520 ${\rm sec}^{-1}$) and low (280 ${\rm sec}^{-1}$) values for the JT8D filter (this averages to 400 ${\rm sec}^{-1}$), the critical deformation rate parameter at $27.6~{\rm kWs/L}$ was $380~{\rm sec}^{-1}$. Occasionally, large variations were observed in the plugging of paper filters that were not seen with metal screens. It is felt that this could be caused by nonhomogeneous filter properties that would be more likely to occur with paper filters than metal screens. In addition to the constant onset with different filters at $27.6~{\rm kWs/L}$, the results in table 3 indicate that $V_c/D_N\varepsilon$ is essentially a constant at higher and lower specific powers (i.e., $V_c/D_N\varepsilon$ is essentially a constant at higher and lower specific powers (i.e., $V_c/D_N\varepsilon$ = 210 and 600 ${\rm sec}^{-1}$ at 20.7 and 34.5 kWs/L, respectively).

An unexpected finding of these experiments was the sensitivity of paper filters to differences between in-line and batch-blended AMK. For example, for slurryblended AMK (degraded at 27.6 kWs/L after 30 minutes) V_c was 0.6 cm/s ($V_c/D_N \epsilon$ = 175 sec^{-1}) with the 80-µm paper. This is one-half of the value that was observed for several different samples of batch-blended AMK shown in table It should be noted that a much smaller difference between in-line blended (age--30 minutes) and batch-blended AMK was observed with metal screens (i.e., V_c in-line blended = 0.8 V_c batch-blended). This finding illustrates that paper filters are more sensitive to minor differences in degradation levels than metal screens. Moreover, when the in-line blended sample was allowed to equilibrate overnight, and then degraded at 27.6 kWs/L, the critical velocity for the 40- μ m filter (JT8D engine) was only 0.08 cm/s ($V_c/D_N \epsilon = 60 \text{ sec}^{-1}$) as compared to 0.36 < V_c < 0.56 cm/s (280 < V_c/D_N ε < 520 sec⁻¹) for batch-blended This low value of 0.08 cm/s was checked by a repeat experiment. Furthermore, after 3 days, the in-line blended AMK was degraded at 34.5 kWs/L and V_c was estimated to be 0.2 cm/s ($V_c/D_N \epsilon$ = 160 sec⁻¹) with the 40- μ m filter, compared to 0.75 < V_c < 0.82 cm/s (580 < V_c/D_N ϵ < 630 sec⁻¹) for batch-blended AMK (table 3). These results run counter to our expectations that the filtration characteristics of in-line blended AMK should approach those of batch-blended AMK; however, they are limited to only one in-line blended sample. Therefore, additional experiments with several different in-line blended samples are needed before any definite conclusions can be drawn regarding the degradation/filtration properties of in-line blended AMK with paper filters.

PHASE II CONCLUSIONS.

Die Swell. The following conclusions concerning die-swell can be made.

- l. Die-swell ratios for AMK change with polymer concentration but the changes are relatively small for $\rm R_e > 100$. Much larger die-swell ratios (4.0 compared to 1.2) could be measured if experiments were conducted at lower Reynolds numbers (i.e., with smaller diameter tubes and at shear rates near the maximum apparent viscosity).
- 2. Normal stresses, calculated from die-swell, are essentially independent of polymer concentration (0.25-0.35 wt percent FM-9). Therefore, they are of little value for quality control. The reason for this is believed to be due to limitations of the current theory that relates die-swell to rheological properties.
- 3. Based on die-swell measurements, in-line blended AMK becomes rheologically equivalent to batch-blended AMK in approximately 30 minutes. While both the video-micrometer and a commercial die-swell tester produce equivalent results, the latter is better suited for real-time quality control.
- 4. The use of differential pressure taps makes it possible to measure the viscosity of AMK without making corrections for viscoelastic end effects. This is particularly important for real-time quality control.

- 5. Thixotropic changes in the flow-induced structure of AMK can be avoided by using relatively short (200 \leq L/R \leq 400) capillary tubes.
- 6. AMK starts to shear thicken at 4V/R near $1000 \, \mathrm{sec}^{-1}$ and reaches its maximum viscosity near $2000 \, \mathrm{sec}^{-1}$. The decrease in viscosity at higher shear rates is due to shear thinning (n' < 1) rather than polymer degradation.
- 7. The maximum apparent viscosity of AMK shows a strong (linear) dependency on polymer concentration, and should be useful criterion for real-time quality control.

<u>Simultaneous Degradation/Filtration.</u> The following conclusions concerning simultaneous degradation/filtration can be made.

Square Mesh Metal Screens.

- 1. For a specific degrader power of $28.6~\rm kWs/L$ (4000 psi), the critical velocity of in-line blended AMK (age--30 minutes) is only slightly less than for the same fuel (age--24 hours) or for batch-blended AMK.
- 2. When critical velocities obtained with different size (44 to 200 $\mu m)$ screens are expressed in terms of a deformation rate (V_c/D_pA_o) , where D_p is screen pore size and A_o is the fractional open area), the onset of plugging, at constant specific power, is 8 x $10^3~\rm sec^{-1}$ for in-line blended (age--30 minutes) and 1 x $10^4~\rm sec^{-1}$ for batch blended.
- 3. There appears to be a lower threshold of specific power (approximately $6.7~\mathrm{kWs/L}$) below which the critical deformation rate is only slightly higher than for undegraded AMK.
- 4. The high filtration ratios for in-line blended AMK that is degraded 30 minutes after blending is caused by a rapid decrease in the critical velocity or deformation rate with the time elapsed after degradation. This effect (particularly in regard to the rate of plugging at the critical) is much more evident with freshly blended (30 minutes) AMK than equilibrated AMK.
- 5. Degradation of in-line blended AMK to meet the high velocities associated with the small flow areas of the fuel-controller wash-flow screen can be achieved within 30 minutes for a specific power <27.6 kWs/L at fuel temperatures near ambient.
- 6. Even at velocities well above the critical (V = $2V_c$), the rate of plugging for AMK degraded at 27.6 kWs/L is so low (0.05 psi/min) that exceeding the critical for short-term operations, such as take-off and landing, should not cause any significant problems.

Paper Filters.

1. The critical velocity of batch-blended AMK (age--30 minutes) degraded at 27.6~kWs/L is much lower (1/20) for a paper filter than for a square mesh metal screen with the same nominal pore size.

- 2. When the critical velocities of AMK (constant specific degrader power) measured with different paper filters are expressed in terms of a critical deformation rate $(v_c/D_N\epsilon$, where D_N is the nominal pore size and ϵ is the porosity), the onset of filter plugging is essentially constant.
- 3. Paper filters are much more sensitive than square-mesh metal screens to subtle differences in the method of blending AMK and the age of the sample at the time of degradation. This sensitivity can be partly explained by the more complex pore structure of paper filters and the longer residence times associated with depth filters compared to surface filters.

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